Year: 2020

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DOI: https://doi.org/10.1590/0103-6440202002884

Originally published at:
Campos, Maria de Fátima T P; Moura, Dayanne Monielle Duarte; Borges, Boniek Castillo Dutra; de Assunção, Isauremi Vieira; Caldas, Marília Regalado Galvão Rabelo; Platt, Jeffrey A; Özcan, M; Souza, Rodrigo Othávio de Assunção E (2020). Influence of acid etching and universal adhesives on the bond strength to dentin. Brazilian Oral Research, 31(3):272-280.
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Influence of acid etching and universal adhesives on the bond strength to dentin

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Short title: Universal adhesives in dentin cavities

Abstract

Purpose: to evaluate the influence of the application mode of three universal adhesive systems on interfacial physical properties of indirect composite restorations adhesively cemented to dentin cavities. Materials and Methods: 78 bovine lower incisors were selected and a slice of dentin (thickness: 2mm) between the buccal surface and pulp chamber was obtained for each tooth. Conical cavities were made on this surface. The internal walls of the cavities were then coated with a hydrophilic gel, filled with composite resin and photopolymerized. The dentin/cone sets were divided into 6 groups (n=10) according to type of universal adhesive (TETRI: Tetric N Bond, FUT: Futura Bond U, SBU: Single Bond Universal) and acid etching on dentin (A: with acid etching; WA: without acid etching). The acid etching and the adhesive systems were applied to the surface of the dentin. All composite resin cones were sandblasted (Al_2O_3, 20s) and silanized. After surface treatment, the cones were cemented (RelyX Ultimate /3M ESPE, St. Paul, USA) into the dentin cavity and photopolymerized. After thermocycling (10,000 cycles) samples were submitted to marginal adaptation analysis (using caries detector dye), push-out test (0.5 mm/min), and failure mode analysis. Additional samples were prepared for nanoleakage analysis (SEM). Results: The data (MPa) were analyzed by two-way ANOVA and Tukey's post-test (5%). The groups in which the dentin was acid etched showed significantly lower bond strength values in the push-out test ($p<0.01$). Conclusions: Dentin acid etching significantly reduced the bond strength between universal adhesive systems and dentin in indirect restorative procedures.

Keywords: Indirect composite resin, Push-out, Universal adhesives, Dentin.

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1. INTRODUCTION

Direct composite resin restorations, as well as indirect or semi-direct1 restorations, have been an option in the dental practice. Indirect and semi-direct techniques overcome some of the disadvantages of direct restorations, such as polymerization contraction and the cementation "gap"(1). In addition, these restorations have better mechanical properties due to the additional polymerization with light or heat, lower microleakage, lower costs, and easier intraoral maintenance compared to dental ceramics (1) Several studies have evaluated the longevity of resins for indirect and semi-direct techniques, reporting good clinical performance in several situations, such as in class I and II restorations (95% in good conditions after 3 years of follow-up) (2), and in inlays (80% after 10 years of follow-up) (3), and class II restorations using the semi-direct technique (100% after 3.5 years) (1).

However, in spite of the excellent longevity, problems related to dentin adhesion have been reported (4). Some factors, such as overdrying of dentin after acid etching, excess moisture, and excessive acid demineralization or acid undercorrosion, may decrease the flow of resinous monomers along the intertubular dentin, compromising the longevity of restorations (5). Although the conventional approach for adhesive restorations, including dentin etching with phosphoric acid (35-37%), is an established and predictable clinical procedure, the acid corrosion of dentin is a definitive factor for adhesion quality, increasing wettability and surface roughness and allowing the penetration of adhesives and resin cements through the smear layer (4).

The universal adhesive systems minimize problems associated to the substrate, ensuring a greater adhesion stability. These materials can be used by the self-etch (SE) technique, prior conditioning etch-and-rinse (ER) technique or as SE adhesives in dentin and ER in enamel (commonly referred to as "selective enamel conditioning") (6). All the components of universal adhesive systems come in a single vial, and the adhesive has the advantages of being effective on wet or dry dentin (7), being less technique sensitive and requiring fewer clinical steps (7). Despite similarities with other adhesive systems, universal adhesives differ from the current SE systems by having phosphate monomers in their composition, among them MDP, which can produce chemical and micromechanical adhesion to dental substrates by ionically binding to calcium in hydroxyapatite (Ca10 [PO4]6 [OH]2) and increasing binding efficiency(6,7).

In addition to interacting with a hydrophilic substrate, the combination of properties allows the interaction with the hydrophobic restorative material under a variety of surface conditions (5).

The adhesion of composite restorations to dental substrates is still a challenge due to the presence of different interfaces: substrate / adhesive / cement system, in addition to the cement/composite resin interface. Moreover, the resin/dentin interface is constantly submitted to mechanical stress from chewing and swallowing (4) and to thermal variations from food. Limitations of materials and techniques may also contribute to a degradation of the tooth/restorative material interface, compromising longevity (8).

Studies have evaluated the bond strength between universal adhesive systems and dentin using direct composites (1). However, no study has evaluated the effects of dentin pretreatment for universal adhesive systems used in the cementation of semi-direct resins to dentin. Thus, the objective of the present study was to evaluate the influence of three universal adhesive systems used with or without acid conditioning on the marginal adaptation, push-out bond strength and nanoleakage of a semi-direct composite resin restoration adhesively cemented to bovine dentine. The hypotheses tested were: a) the type of adhesive system affects bond strength; B) acid conditioning does not significantly affect bond strength; C) the adhesive system and the acid conditioning technique will not affect the marginal adaptation or the nanoleakage.

2. MATERIAL AND METHODS

The materials (manufacturers, trademarks, chemical composition and batch number) used in this study are presented in Table 1.

2.1. Teeth selection and preparation

Samples from this study were prepared according to a method described previously for the push-out bond strength test (9). Seventy-eight intact bovine incisors were selected, cleaned from tissue and debris with a periodontal curette, disinfected with 0.1% aqueous thymol solution at 40°C for one week, and stored in distilled water at 4°C (ISO 11405). The roots were sectioned at the cementoenamel junction with a double-sided diamond disk (KG Sorensen, Barueri, SP, Brazil) in a straight handpiece and low speed micromotor, under constant irrigation. (Fig 1A).

A slice of dentin (thickness: 2mm), between the buccal surface and pulp chamber, was obtained for each tooth. The teeth were ground with # 200, # 400 and # 600 grit sanding paper, in a polishing machine (AROTEC, Cotia, SP, Brazil), and the thickness measured with digital caliper (Fig. 1B and 1C). “Then, standardized 3D dentin conical cavities (larger Ø: 2 mm, smaller Ø: 1.5 mm) were prepared using tapered diamond burs (#3131, KGSorensen, Barueri, SP, Brazil) in a high-speed handpiece adapted to a dental surveyor (Fig. 1DE), so that the active tip was perpendicular to the buccal surface of the disc; the perforation was done in a single lowering movement by a single operator (Fig. 1D-E). The diamond bur was replaced after preparation of 50% of the samples.

2.2. Preparation of composite resin cones

Opallis composite resin (FGM, Joinville, SC, Brazil) was used to fill the 3D dentin preparations. The dentin samples were individually positioned on a glass surface and a hydrophilic gel (K-Y Gel Johnson & Johnson, New Jersey, USA) was applied to the internal walls of the cavities with a microbrush, which were filled with a single increment of composite resin (2 mm) (Fig 1F) and partially photopolymerized for 3 s on each side to allow removal of excess resin (Fig 1G). Resin cones were then removed from the perforation and subjected to final polymerization for 40 s using a LED light (Radii-Cal - SDI 1200 mW/cm2) (Fig 1H). “Afterwards, polymerization of the resin cones was further complemented in a microwave oven for 3 minutes at maximum power (Fig. 1I). The hydrophilic gel from the internal walls of the 3D dentin cavities was removed with an air/water spray. Dentin/composite cone sets were stored for 24 hours in distilled water at room temperature and then finished and polished with Sof-lex discs (3M ESPE, St. Paul,MN, USA).The sets were randomly divided into 6 groups (n = 10) according to the "adhesive system" factor (3 levels), and "acid conditioning" factor (2 levels, with and without): Futura Bond U (FUT); acid+FUT (A+FUT); Scotchbond Universal Adhesive (SBU); A+SBU; Tetric N-Bond Universal (TETRI); and A+TETRI.

2.3. Cementation techniques

2.3.1.Surface treatment of composite resin cones

Cones were cleaned in an ultrasonic bath (Cristófoli, Campo Mourão, Paraná, Brazil) with 10% isopropyl alcohol for 5 minutes, which were sandblasted with 50 µm aluminum oxide particles for 20 seconds (2.5 bar), slope of 90°, at a distance of 10 mm from the surface bonding. Using a microjet device (Microjato Standard, Bioart, São Carlos, SP, Brazil) attached to a dental surveyor (Fig 1J), the cones were rotated during the air-abrasion, so that only the bonding surfaces were sandblasted. After treatment, the surfaces were again cleaned in ultrasonic bath with distilled water for 2 minutes and air-dried. A layer of Silane (Dentsply, Pennsylvania, USA) was applied to the sandblasted surfaces of the cones with the aid of a microbrush (Dentsply, Pennsylvania, USA), according to manufacturer's recommendation.

2.3.2. Dentin Surface Treatment

Prior to cementation, prophylaxis in the cavity of the dentin surface was performed with pumice stone and water using a Robinson mini brush (Ø: 1.5mm) (microtuft- Dhpro – Paraguaná, PR, Brazil) at low speed. Samples were then washed with water-air jet for 30 s and the excess moisture removed with absorbent paper. In three groups, the adhesive systems were directly applied according to the experimental group. In the other groups, the dentin surface was conditioned with 37% phosphoric acid (Dentsply Conditioner) for 15 s, carefully washed with water jet for 30 s (Fig 1K) and partially dried with absorbent paper. The adhesive systems were applied according to the manufacturer's recommendations (Fig 1 KL):

• SBU: one layer was actively applied for 20 s, followed by a light jet of air for 5 s for solvent evaporation, and photopolymerization for 10 s using a LED light curing device (1200 mW/cm2) (Radii Cal, SDI, Australia).

• FUTURA BOND U: One layer of the adhesive was actively applied for 20 s, followed by a light air jet for 5 s and photopolymerization for 10 s using a LED light curing device (1200 mW/cm2) (Radii Cal, SDI, Australia).

• TETRIC N BOND: One layer of the adhesive was actively applied for 20 s, followed by a light air jet for 5 s and photopolymerization for 10 s using a LED light curing device (1200 mW/cm2) (Radii Cal, SDI, Australia).

2.4. Cementation of resin cones

The dual resin cement RelyX Ultimate (3M-ESPE, Minnesota, USA) was used for the cementation of cones. The dentin samples were placed individually on a glass plate. Equal amounts of base and catalyst pastes were dispensed, mixed, and immediately applied to the cementation surface of the cone, which was positioned into the dentin cavity (Fig 1M). The top of the cone was covered with a polyester strip and a 750 g weight was applied, simulating the adhesive cementation protocol (Fig 1N). Both sides of the restoration were light cured for 40 s using a LED device (1200 mW / cm2) (Radii Cal, SDI, Australia). The surfaces were then polished with polishing systems Sof-Lex Pop-On (3M-ESPE, Minnesota, USA). The specimens were stored in distilled water at 37°C for 24 h and then submitted to a thermocycling aging protocol of 10,000 cycles of alternating 30 s baths of 5 and 55°C, with a 2 s interval between immersions.

2.5. Marginal adaptation

To determine the marginal adaptation, a dye technique was used (10). After thermocycling, previously to the push-out test, all samples were submitted a 1% solution of red propylene glycol (caries detector dye, Kuraray Co., Osaka, Japan) was applied to the margins of the restoration for 5 s. Specimens were then rinsed in tap water and gently dried. They were then attached vertically to a holding devise coupled with a lateral ruler of 2 cm, allowing calibration. Subsequently, photographs of the top and bottom of the restoration were obtained with a Canon EOS Rebel T5i Camera, positioned at a focal distance of 60 cm. The amount of stained margins was analyzed using Image Pro-Plus 7.0 (Media Cybernetic) software. The perimeter of the cementation line was measured in the photographs of each sample using a micrometric scale, which allowed following the contour of the restoration. The stained areas in the margins were then measured in both sides of each sample. This technique stained the gaps so they could easily be quantified (10). Data was entered in an EXCEL spreadsheet to calculate the percentage of gaps in each sample and analyzed statistically. The marginal adaptation evaluation was done by single trained evaluator. After, the all samples were submitted to push- out test.

2.6. Push-out bond strength test

The push-out bond strength test was performed using a universal testing machine (model 4411; Instron Corp., Canton, MA, USA). A metal device with a central hole (Ø: 1mm) was adapted to the base of the machine. Specimens were placed in the device with the larger diameter in contact with the lower metal surface of the device and the smaller diameter in contact with the metal piston (Ø: 1 mm). A 50 KgF load cell was positioned at the center of the composite resin cylinder at a speed of 0.5 mm/min until failure (Fig. 10). The load required for failure was recorded by the test machine and subsequently converted to MPa values. The resistance values were calculated (in Mpa) by dividing the force (in N) at time of failure by the area: p (R1 + R2) H (R1 + R2) 2 + h2, where R1 represents the smaller radius, R2 is the larger radius and h the height of the cavity.

2.7. Failure mode analysis

Specimens were examined by stereomicroscopy (20×) (Stereo Discovery V20, Zeiss, Göttingen, Germany) and failure modes were classified with the following scores: AD, adhesive failure between dentin and cement; AR, adhesive failure between cement and composite resin; C1, cohesive failure in dentin; C2, cohesive failure in composite resin; C3, cohesive failure in cement and mixed failures: (Cohesive + AR or AD).

2.8. Nanoleakage (NL)

For nanoleakage analysis, three extra samples from each group were made, following the same parameters. After thermocycling (10.000 TC) the samples were immersed in distilled water and stored for 24 hours at 37°C in an oven. The specimens were then removed from the water, dried with absorbent paper and impermeabilized with two layers of nail polish (Colorama, CEIL, São Paulo, SP, Brazil), leaving a 1 mm space from the edge without nail polish. The specimens were immersed in distilled water for 20 minutes and subsequently in silver nitrate solution (prepared with 25 g of silver nitrate crystals; Sigma Chemical Co., St. Louis, MO, USA), pH = 11.0, in a dark container for 24 hours. Afterwards, specimens were washed with distilled water and immersed in Kodak Developer Solution (Carestream Health Inc. NY, USA) for 8 h under fluorescent light. Again, specimens were washed in distilled water and polished under water in a polishing machine (Metaserv 2000, USA) using #600, 1200, 2000 grit sand paper (Carbimet Disc Set, USA) and 0.3 µm and 1 µm polishing pastes (Alumina Polishing Abrasives - PACE Technologies, Tucson, USA) using a felt disc (Buehler, UK, USA). Samples were examined in SEM / EDS (JEOL-JSM 5600LV, Tokyo, Japan) at 600, 800 and 1000× increments. Silver penetration at the bonding interface, the hybrid layer, and adhesive layer were examined by a single evaluator.

2.9. Statistical analysis

The bond strength data were submitted the test of normality Kolmogorov-Smirnov using the computer program Assistat 7.7. The results indicated normally distributed ($p>0,05$) and the parametric tests of two-way analysis of variance (ANOVA) and Tukey's post-test (5% significance level) in the program Assistat 7.7. Failure modes and complementary data were analyzed descriptively. The marginal adaptation data were submitted to three way ANOVA, followed by Mann-Whitney test and t-test (5% significance level). Data from nanoleakage were descriptively analyzed.

3.0. RESULTS

3.1. Push-out bond strength

The interaction between factors (adhesive system × acid conditioning) was non-significant ($p = 0.514$). The "adhesive system" factor ($p = 0.532$) did not present a significant effect on results. On the other hand, the "acid conditioning" factor ($p = 0.0001$) was statistically significant. The results of the push-out test and the comparison between groups are shown in Table 2. Acid-etching the dentine prior to cementation significantly decreased bond strength of the three adhesive systems compared to no conditioning;

3.2. Marginal adaptation

In general, no significant difference in the percentage of stain infiltration was found between presence and absence of acid etching ($p>0.05$, Tukey test); the exception was in the FUT groups, where the acid etching group showed significantly more infiltration than the acid-free groups. Significant differences were also found for the acid-free SBU group in the larger diameter region, which showed significantly greater marginal infiltration than the acid-free groups. The smaller diameter region showed a significantly higher infiltration in the acid groups of the FUT and TETRI adhesive systems. Between groups comparisons are presented in Table 3.

3.3. Nanoleakage

Different patterns of silver nitrate nanoleakage were found along the adhesive layer for the three universal adhesive systems. The deposition of silver ions was found throughout the adhesive layer at the cement/dentin interface. The TETRI -A group (Figure 2E) demonstrated a thicker layer of silver ions compared to the non-conditioned group (Figure 2F). SEM images showing silver particles in the adhesive systems are shown in Figures 2 A-F. Elemental silver was identified by EDS analysis, confirming the obtained results.

3.4. Failure analysis

Different failure patterns were observed for the three adhesive systems tested according to absence or presence of acid conditioning. The groups with acid etching demonstrated mixed failures (cohesive in cement and adhesive at the cement/dentin interface) (Figure 3A). In groups without acid etching, failures occurred mainly at the cement/resin interface (Figure 3B). Adhesive failures at the cement/dentin interface were common for the SBU-A and TETRI-A groups (Figure 3 C). Cohesive failures in dentin were also observed in the SBU and TETRI groups (Figure 3D). Failure modes for each group are shown in table 4.

4.DISCUSSION

In the present study, the influence of acid etching on the bond strength between three universal adhesive systems and bovine dentin was investigated. In this study, cavities in coronary bovine dentin was standardized at 2 mm thickness to simulate a clinical situation of high C-factor, similar to a semi-direct composite resin restorations performed clinically, besides allowing the evaluation of marginal adaptation, bond strength and nanoleakage in the same cavity (11,12).

According to results of this study, the first hypothesis that the adhesive system influences bond strength was not accepted. In this study, no significant difference between the three universal adhesives was found. Tetric N Bond, SBU and Futura Bond U are universal one-component adhesives with similar indications, applications, and chemical compositions. All have phosphated acid monomers in their composition and are considered 'ultra mild' based on their pH (SBU: pH=2.7; FTU: pH=2.5 and TET: pH=2.5–3.0), (13,1) which makes them capable of demineralizing and diffusing in the dentin, forming a hybrid layer more stable to hydrolytic degradation due to changes in their chemical composition (11). The phosphated functional monomers in their composition chemically interact with hydroxyapatite forming hydrolytic, forming more stable bonds with calcium (14,15). It is reported that adhesive systems containing MDP phosphated monomers chemically interact with hydroxyapatite forming 10-MDP-Ca salts that have low solubility, better resistance to hydrolysis, and are more stable. However, in this study, the adhesive systems that contained this monomer (SBU and TETRIC) did not significantly influence the bond strength (16).

A recent study (17) evaluated the bond strength of the resin/dentin interface using two of these systems (SBU and Tetric N Bond) and the authors found no difference between them. The author reports that the interfacial morphology of both adhesives can be affected by the similarity of their compositions, as both contain water, ethanol, and hydroxyethyl methacrylate (HEMA). Water is essential to ionize acid monomers and trigger the demineralization process (13). The water-ethanol combination can also dilute the viscous monomers and help their infiltration into the dentin. In another study (18), the authors report that Futura Bond U (FUT) presented significantly higher values of bond strength compared to other universal adhesives, such as Clearfil Universal and SBU, and they associate the results to a greater interaction of FUT with the resin.

The second hypothesis tested in this study that acid etching does not significantly affect bond strength was not accepted. The universal adhesives are known for their versatility and by being effective either with or without prior acid conditioning. Thus, is expected that bond strength would not be compromised by acid conditioning (19). However, prior acid etching of dentin significantly decreased the adhesion values of the three universal adhesive systems. According to the concept of adhesion-decalcification (A-D) proposed for self-adhesives (19), dentin demineralization by strong acids will result in a higher dissolution rate of calcium salts. This suppresses the potential of establishing a chemical bond between resinous monomers and apatite crystals, and creating calcium precipitates nano-layers with phosphate monomers (20). When the conditioning and simultaneous infiltration of adhesive systems into the dentin, as proposed by the universal adhesives, is replaced by a diffusion mechanism to achieve micromechanical retention (as in acid corrosion) (21), an incomplete infiltration of resinous monomers within a matrix of thickened or completely demineralized collagen may occur.

Varied results are found in the literature (19,21,15). One study (22), reported that dentin conditioning had no negative impact on adhesion. Corroborating these findings, another study (2), concluded that the prior acid etching of dentin did not significantly affect bond strength of two universal adhesive systems, Futura Bond U and SBU. The authors reported that the additional application of acid monomers on dentin surface enhanced by the active friction of the adhesive system seems to improve the contact area of the adhesive solution on the surface and provide a higher concentration of free H+ ions to interact with the mineral components of dentin (22,2). Additional studies (22), also found no difference between universal adhesives (All-Bond Universal, Scotchbond Universal, and Futura Bond U) when used with different conditioning techniques. On the other hand, one study (23), reported that prior acid etching reduced dentin bond strength values only for some of the universal adhesive systems tested, such as Futura Bond U, but stated that universal adhesives have specific application methods and that acid pretreatment should be performed only on enamel. Another relevant factor that was accounted for in this study is the perforation simulating a clinical situation, as the adherent surface of the mineralized dentin depends on cavity configuration (Factor C), that is, the option of pre-conditioning is determined based on cavity size and depth (23).

The third hypothesis that acid conditioning does not affect marginal adaptation or the nanoleakage was partially accepted. Was used in this study the caries detector stain analyses to evaluate the marginal adaptation. Based on a study (10), measuring the margin gaps using the staining technique provides results comparable to scanning electron microscopy. According results of this study, acid etching, especially for Futura Bond U groups, significantly increased the percentage of gaps in relation to the acid-free groups. On the other hand, SBU groups showed significantly higher amount of gaps in the acid-free groups. Larger stained areas around the margins of the restoration, indicating higher percentage of gaps (10), are the first sign of failure of a restoration, clinically detectable by marginal coloring.

With regard to Nanoleakage, SEM images and EDS analysis demonstrated the infiltration of silver ions along the adhesive layer (dentin/adhesive interface), was assigned for samples with and without acid conditioning of the three adhesive systems. However, for the TETRI + acid conditioning group, a thicker hybrid layer with a higher concentration of silver ions along the adhesive interface was observed compared to TETRI (without acid conditioning) samples. Silver nitrate can lodge into nanometer-sized spaces around exposed collagen fibers where monomers failed to infiltrate or where residual water was not displaced by the adhesive or even in areas with incomplete monomer conversion (9), which are important factors for degradation of the bonding interface. Adhesive systems that contain both MDP and HEMA, such as Tetric N-Bond, may create interfaces bound for nanoleakage, as monomers compete for the interaction with calcium on the dentin surface, resulting in markedly reduced nano-layering of 10-MDP-calcium salts within the resin-dentin interface (24). In addition, thermocycling can accelerate aging degradation and cause expansion and tension stresses due to the different thermal expansion coefficient between substrates and restorative materials, favoring interface degradation (16).

Failure mode analysis demonstrated different failure patterns between acid and acid-free groups. In general, failures were of mixed mode. The groups with acid conditioning showed inferior adhesion at the cement/dentin interface because they presented a higher rate of mixed failures. The opposite was observed for acid-free groups. The process of nucleation is the failure of materials or interfaces, i.e., it refers to weak points where high stresses can lead to overload. Acid etching of dentin prior to using universal adhesives creates weak regions in the interface between resin and the adhesive layer or between the adhesive layer and decalcified dentin (24). In addition, such vulnerable regions and flawed bonding may adversely affect long-term adherence (22).

The results of this study suggest that the three universal adhesive systems presented similar performance. However, acid conditioning of dentin significantly reduced bond strength of the adhesives used in semi-direct composite restorations. The marginal adaptation demonstrated that groups with acid etching, especially for Futura Bond U, significantly increased the percentage of gaps compared to the acid-free groups, and with regard to nanoleakage, the three adhesive systems demonstrated infiltration of silver ions along the adhesive layer with and without acid conditioning.

Moreover, further controlled and randomized studies are required to evaluate adhesion and longevity of universal adhesive systems and to complement these laboratory findings.

RESUMO:

Objetivo: avaliar a influência do modo de aplicação de três sistemas adesivos universais nas propriedades físicas interfaciais de restaurações indiretas de compósito adesivamente cimentadas a cavidades dentinárias. Materiais e Métodos: 78 incisivos inferiores bovinos foram selecionados e uma fatia de dentina (espessura: 2mm) entre a face vestibular e a câmara pulpar foi obtida para cada dente. Cavidades cônicas foram feitas nesta superfície. As paredes internas das cavidades foram então revestidas com um gel hidrofílico, preenchidas com resina composta e fotopolimerizadas. Os conjuntos dentina / cone foram divididos em 6 grupos (n = 10) de acordo com o tipo de adesivo universal (TETRI: Tetric N Bond, FUT: Futura Bond U, SBU: Single Bond Universal) e ácido na dentina (A: com ácido gravura; WA: sem condicionamento ácido). O condicionamento ácido e os sistemas adesivos foram aplicados na superfície da dentina. Todos os cones de resina composta foram jateados (Al_2O_3, 20s) e silanizados. Após o tratamento superficial, os cones foram cimentados (RelyX Ultimate / 3M ESPE, São Paulo, EUA) na cavidade dentinária e fotopolimerizados. Após a termociclagem (10.000 ciclos), as amostras foram submetidas à análise de adaptação marginal (usando corante detector de cárie), teste push-out (0,5 mm / min) e análise do modo de falha. Amostras adicionais foram preparadas para análise de nanoinfiltração (MEV). Resultados: Os dados (MPa) foram analisados por ANOVA two-way e pós-teste de Tukey (5%). Os grupos em que a dentina foi condicionada por ácido apresentaram valores significativamente menores de resistência de união no teste push-out ($p < 0{,}01$). Conclusões: A gravação com ácido dentinário reduziu significativamente a força de união entre sistemas adesivos universais e dentina em procedimentos restauradores indiretos.

CONFLICTS OF INTEREST

The authors report no conflicts of interest.

ACKNOWLEDGEMENT:

"This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001".

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Table 1. Trademarks, manufacturers, chemical composition and batch number of the materials used in the study.

Trademark	Type	Manufacturer	Chemical composition	Batch (nº)
Single Bond Universal	Adhesive System	3M/ESPE, EUA	MDP (1-10%), dimethacrylate (15-25%), HEMA, vitrebond copolymer (1-5%), filer, etanol (25-30%), water (10-15%), initiators, and silane (5-15%)	1513900170
Tetric N Bond Universal	Adhesive System	Ivoclar Vivadent/Brazil	BisGMA (25- 50%), Water and Ethanol (10-<25%), 2-hydroxyethyl methacrylate (HEMA) (10-<25%), Phosphonic acid methacrylate (MDP) (10-<25%), Diphenyl (2,4,6- trimethylbenzoyl) phosphine oxide (1-<2.5%) Urethane dimethacrylate (0.3-<10)	V11838
Futurabond U	Adhesive System	Voco, Germany	Liquid 1: HEMA (25-50%), Bis-GMA (25-50%), HEDMA (10-25%), acidic adhesive phosphate monomer (5-10%) Urethane dimethacrylate (5-10%), catalyst (<2.5%). Liquid 2: etanol (50-100%), Initiator (2.5-5%) catalyst (1-2 – 5%)	1519237
RelyX Ultimate	Resin cement	3M ESPE/ EUA	Silane treated glass powder (50-60%) 2-propenoic acid (2-methyl-,1,1-[1-(hydroxymethyl)-1, 2-ethanediyl]- ester, reaction products with 2-hydroxy-1,3-propanediyl DMA and phosphorus oxide (20-30%), TEGDMA(10-20%), silane treated sílica (1-10%), oxide glass chemicals (<3%), sodium persulfate (<1%), tert-butyl peroxy-3,5,5-trimethylhexanoate (<0.25%), copper (2+) acetate monohydrate, acetic acid (<0.1%)	1625600718
Phosphoric acid (37%)	Dental conditioning gel 37%.	Dentsply/Brazil	Phosphoric acid, colloidal silica, Surfactant, and pigment.	0564488H
Opallis	Macrohybrid composite resin: (A2)	FGM/ Brazil	Bis-GMA monomers (bisphenol A diglycidildimethacrylate 6-8%) BisEMA (ethoxylated bisphenol A diglycidildimethacrylate 5-10%), TEGDMA (<5%) (triethylene glycol dimethacrylate), UDMA (5-10 urethane dimethacrylate), canphorquinone (<1), co-initiator e silane (5-10%), silanized ceramic (65-75%), pigments e silica.	071215

Table 2 – Means (SD) of the push out strength values in the studied groups

Adhesive system	Acid conditioned	No acid-conditioned
Tetric N Bond	5.54(3.5)$_{bB}$	12.67(6.3)$_{aA}$
Futura Bond	6.34(2.9)$_{bB}$	10.70 (4.2)$_{aA}$
Scotch Bond Universal	5.09(1.9)$_{bB}$	10.22(3.9)$_{aA}$

Upper case letters: comparisons between columns in the same lines.
Lower case letters: comparisons between lines in the same columns.

Table 3. Medians (Q1-Q3) of infiltration percentage of the three adhesive systems in both sides of the specimen.

Side	Adhesive system	Acid conditioned	No acid-conditioned
Upper side	Futura	5.2 (0-31.7)Aa*	0 (0-1.7)Ab
	SBU	4.6 (0-18.3)Aa	12.85 (11.6-20.8)Aa
	Tetric	0.1(0-6.8)Aa*	0(0-13.75)Ab
Lower side	Futura	21.2(20-33.6)Aa	0(0-0.8)Ba
	SBU	6.6 (0-28.6)Aa	15.0 (0-25.8)Aa
	Tetric	24.1 (0 – 35.7)Aa	9.6 (0-17.6) Aa

Upper case letters: comparasion between with and without acid conditioning, in the same acid, and same side.
Lower case letters: comparasion between different adhesive system, in the same side and with the same acid protocol (acid conditioned or no acid-conditioned).
*significant differences between different sides.

Table 4: Number (N.) and percentage (%) of pre-test failure (PTF) during thermal aging, total number of samples submitted to the push-out bond strength test and failure mode (%) of the groups after bond strength test.

Adesive/acid (A) or no acid (WA)	Number of samples	N. and% of spontaneous PTF during aging	N. and % of tested samples	Percentage by failure mode						
				AD	AR	C1	C2	C3	Mixed	Total
Fut/A	10	0 (0)	10(100)	2	-	-	-	-	8	100%
Fut/WA		0(0)	10 (100)	-	-	-	-	-	10	100%
Sbu/A	10	0(0)	10 (100)	3	-	-	1	-	6	100%
Sbu/WA	10	0 (0)	10 (100)	-	-	-	-	-	10	100%
Tetri/A	10	0	10(100)	-	-	-	1	-	9	100%
Tetri/WA	10	0	10 (100)	-	-	-	-	-	10	100%

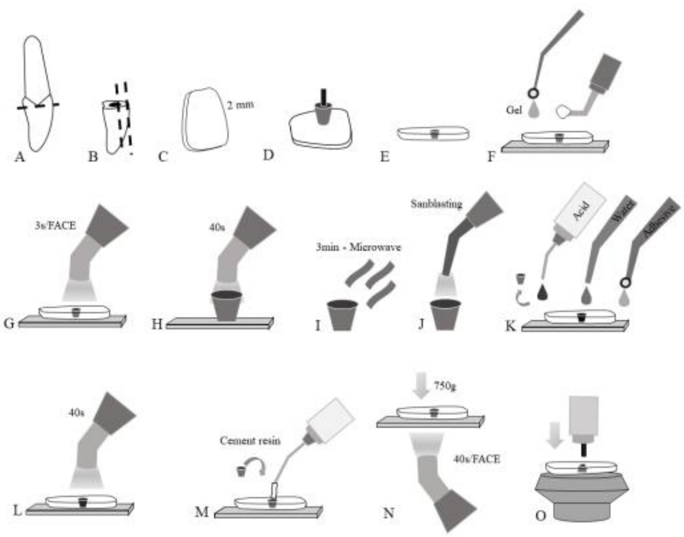
AD = adhesive failure between dentin and cement; AR: Adhesive failure between cement and composite resin C1 = cohesive failure in dentin; C2 = cohesive failure in composite resin; C3 = cohesive failure in cement; Mixed: (Cohesive + AR or AD).

FIGURE LEGENDS:

Figure 1: Sample preparation for the push-out test.

Figure 2: Representative EDS / SEM (200- 1000X) micrograhs of nanoleakage at the cement / dentin / resin interface after adhesive universal with acid and no acid conditioning. A)Futura Bond U with acid; B) Futura Bond U without acid; C) Scotch Bond Universal (SBU) was applied with acid; D) SBU without acid; E) Tetric N-Bond Universal with acid; F) Tetric N-Bond Universal without acid. Δ: Dentin; *: composite resin ➡ : silver ions at the interface, ◊: resin cement.

Figure 3: Stereomicroscopy (20x) micrograph representing failure modes: A) cohesive failure in the cement and adhesive failure at the cement / dentin interface; B) cohesive failure in the cement and adhesive failure at the cement / resin interface; C) adhesive failure at the cement / dentin interface; D) cohesive failure in the dentin and adhesive failure at the cement / dentin interface. Right: Cilynder of composite resin; Left: dentin.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Gel
A
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C
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3s/FACE
40s
3min - Microwave
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Acid
Water
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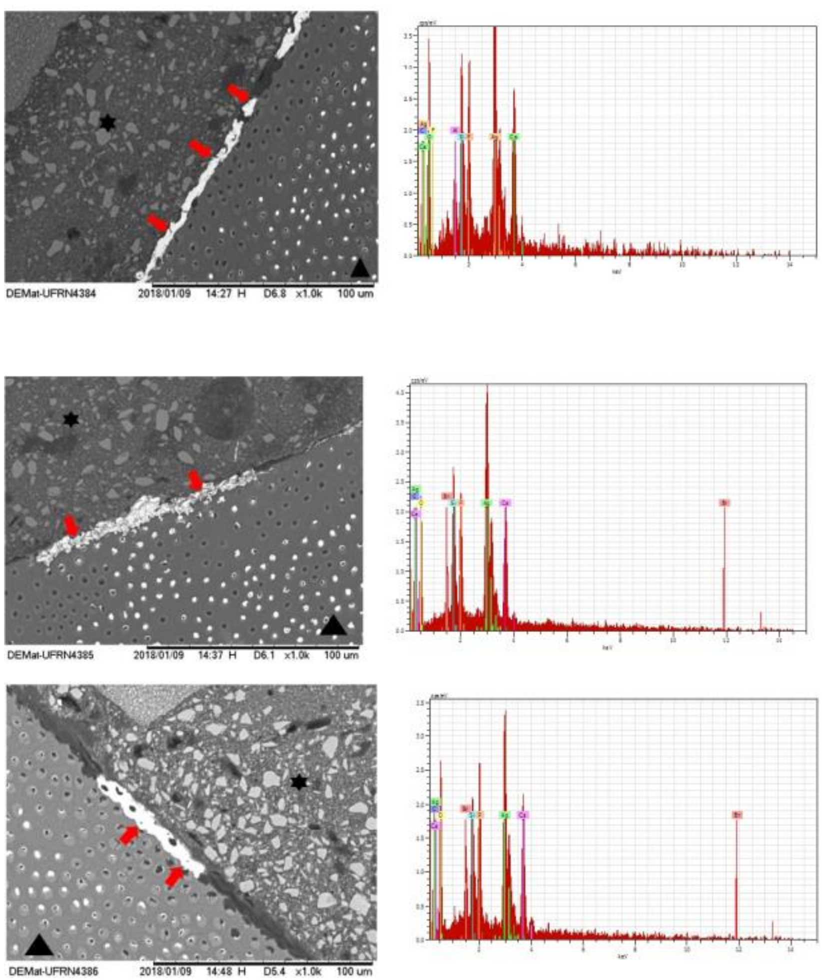
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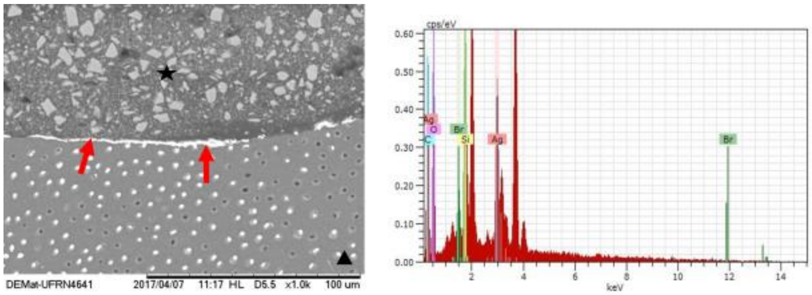
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Si
Ag
Br
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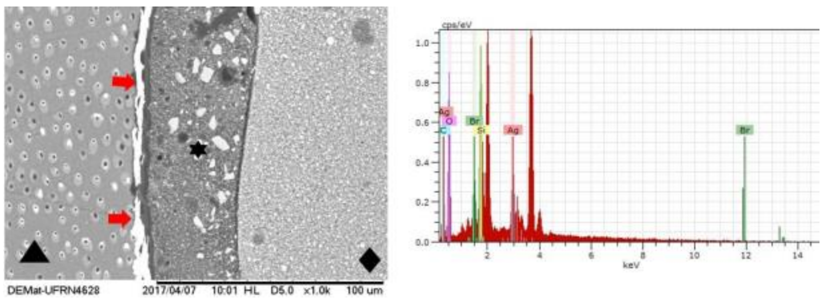
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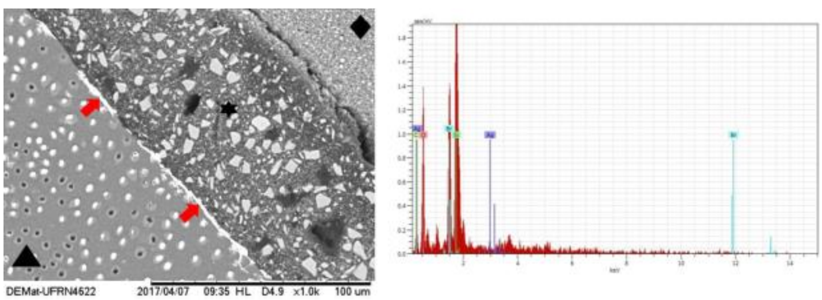
Ag
O
C
Br
Si
Ag
Br
keV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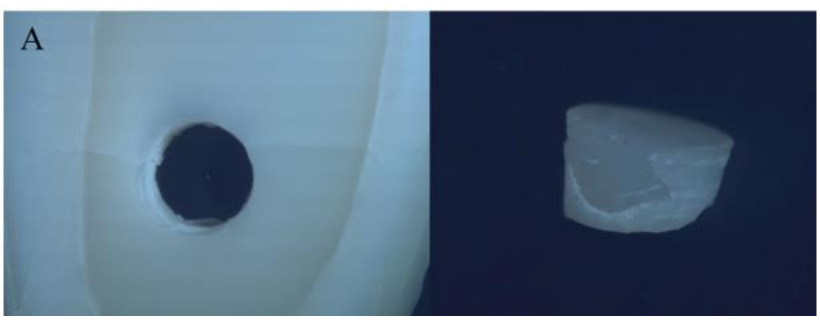
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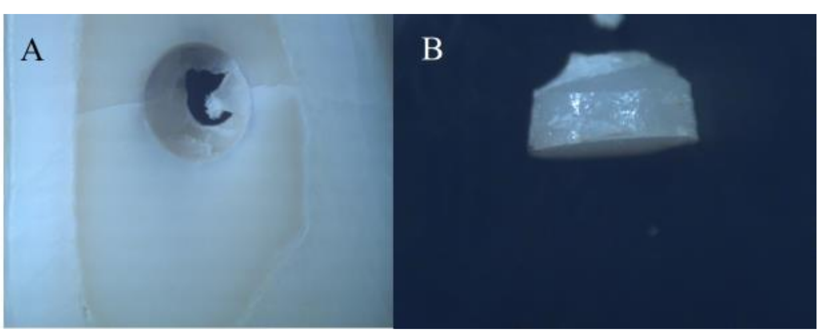
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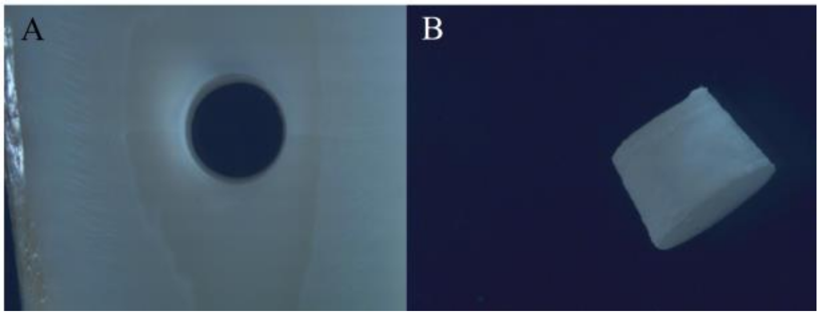
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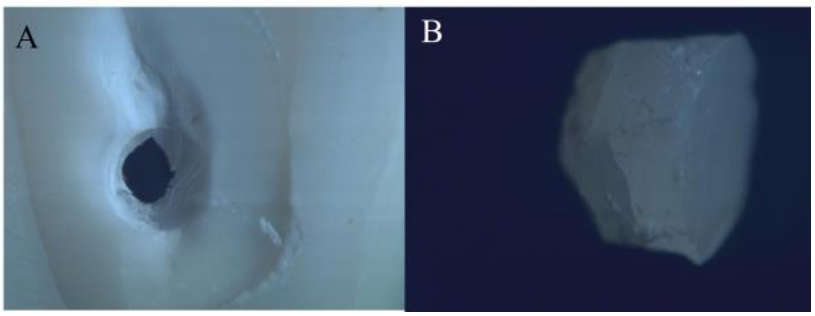
A
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